

DPD Rapid Liquid Method<sup>1</sup>

Method 10059

(0.02 to 2.00 mg/L)

Pour-Thru™ Cell

**Scope and Application:** For treated water.<sup>1</sup> Adapted from *Standard Methods for the Examination of Water and Wastewater*.**Test preparation****How to use instrument-specific information**

The [Instrument-specific information](#) table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

**Table 98 Instrument-specific information**

| Instrument                | Pour-thru Kit   | Cell orientation                                          | Adapter    |
|---------------------------|-----------------|-----------------------------------------------------------|------------|
| DR 6000                   | LQV175.99.20002 | Arrow faces right                                         | —          |
| DR 5000                   | LZV479          | —                                                         | —          |
| DR 3900                   | LQV157.99.10002 | Align cell flow arrows with arrows on cell compartment    | —          |
| DR 3800, DR 2800, DR 2700 | 5940400         | 1-inch (round) path in line with the arrow on the adapter | LZV585 (B) |

**Before starting the test:**

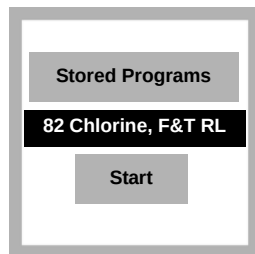
|                                                                                                                             |
|-----------------------------------------------------------------------------------------------------------------------------|
| Analyze samples immediately. Do not preserve for later analysis.                                                            |
| Refer to the instrument User Manual for Pour-Thru cell and module assembly and installation.                                |
| Protect the Pour-Thru Cell from contamination when not in use by inverting a small beaker over the top of the glass funnel. |
| The indicator reagent must be prepared in advance. See <a href="#">Reagent preparation</a> .                                |
| Make sure the pour-thru cell is completely seated in the sample cell compartment.                                           |

**Collect the following items:**

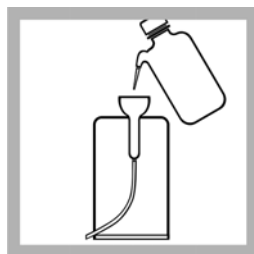
| Description                                                                           | Quantity |
|---------------------------------------------------------------------------------------|----------|
| DPD Indicator Powder                                                                  | varies   |
| Free Chlorine Indicator Solution                                                      | 1 mL     |
| Free Chlorine Buffer Solution                                                         | 1 mL     |
| Cylinder, glass, mixing, 100-mL                                                       | 1        |
| Dispenser, Adjustable Volume                                                          | 2        |
| Pour-Thru Cell Module and Cell (see <a href="#">Instrument-specific information</a> ) | 1        |

See [Consumables and replacement items](#) for reorder information.

### Free Chlorine, rapid liquid method



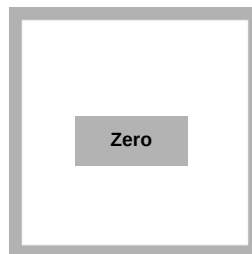
1. Select the test.  
(See [Instrument-specific information](#)).



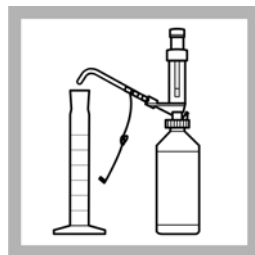
2. Install the Pour-Thru Cell module and cell. See [Instrument-specific information](#) for adapter and cell orientation.



3. Pour approximately 50 mL of sample into the Pour-Thru Cell.



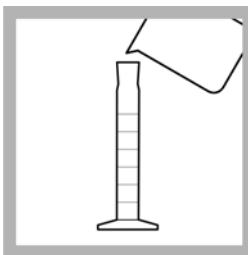
4. When the flow stops, press **ZERO**. The display will show: 0.00 mg/L  $\text{Cl}_2$ .



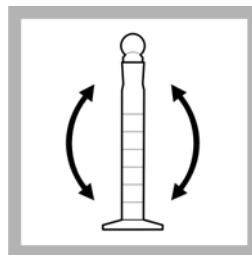
5. Add 1.0 mL of Free Chlorine Buffer Solution to a clean, dry 100-mL glass mixing cylinder using the Dispenser.



6. Add 1.0 mL of prepared Free Chlorine Indicator Solution to the same mixing cylinder using the dispenser. Swirl to mix the reagents. Proceed to step 7 immediately.



7. Carefully fill the mixing cylinder to the 80-mL mark with sample.



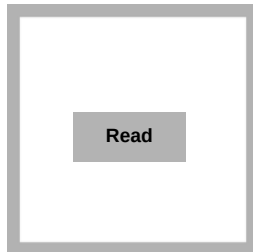
8. Stopper the cylinder and gently invert it twice to mix. Proceed to step 8 immediately.

## Free Chlorine, rapid liquid method (continued)



9. Fill the funnel of the Pour-Thru Cell with the reacted sample from the mixing cylinder.

(It is not necessary to pour the entire sample into the Pour-Thru Cell; approximately half of the sample may be discarded.)



10. After the flow stops, press **READ**. Results will appear in mg/L  $\text{Cl}_2$ .



11. Flush the Pour-Thru Cell with at least 50-mL of deionized water immediately after use.

## Reagent preparation

The Free Chlorine Indicator Solution must be prepared before use. Using a powder funnel, add the contents of one 24 g bottle of DPD Powder\* to one 473-mL bottle of Free Chlorine Indicator Solution\*. Invert several times and swirl until the powder is completely dissolved. A pale pink color may develop, but should not affect results.

This solution will give accurate results for at least one month after mixing when stored at 20–25 °C (68–77 °C). Write the date of preparation on the Indicator Solution Bottle. Discard any remaining solution after one month. Use of this reagent after one month may result in high reagent blanks and low values at high concentration. Do not combine fresh reagent with previously mixed reagent.

## Interferences

Table 99 Interfering substances

| Interfering substance  | Interference level                                                                                                                                                                                                                                                                                    |
|------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Alkalinity             | Greater than 400 mg/L $\text{CaCO}_3$ . May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sulfuric Acid <sup>1</sup> . Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition. |
| Bromine, $\text{Br}_2$ | Interferes at all levels.                                                                                                                                                                                                                                                                             |
| Hardness               | Levels below 1000 mg/L as $\text{CaCO}_3$ will not interfere.                                                                                                                                                                                                                                         |
| Iodine, $\text{I}_2$   | Interferes at all levels.                                                                                                                                                                                                                                                                             |

\* See [Required reagents](#).

Table 99 Interfering substances (continued)

| Interfering substance                                                                                  | Interference level                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
|--------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Manganese, oxidized ( $\text{Mn}^{4+}$ , $\text{Mn}^{7+}$ ) or Chromium, oxidized ( $\text{Cr}^{6+}$ ) | <ol style="list-style-type: none"> <li>1. Adjust sample pH to 6–7 with 1.000 N Sulfuric Acid<sup>1</sup>.</li> <li>2. Add 9 drops Potassium Iodide (30 g/L)<sup>1</sup> to an 80-mL sample.</li> <li>3. Mix and wait 1 minute.</li> <li>4. Add 9 drops Sodium Arsenite<sup>1, 2</sup> (5 g/L) and mix.</li> <li>5. Analyze the treated sample as described in the procedure above.</li> <li>6. Subtract the result of this test from the original analysis to obtain the correct concentration.</li> </ol> |
| Monochloramine ( $\text{NH}_2\text{Cl}$ )                                                              | Samples containing monochloramine will cause a gradual drift to higher chlorine readings. When read within one minute of reagent addition, 3.0 mg/L monochloramine will cause an increase of less than 0.1 mg/L in the free chlorine reading.                                                                                                                                                                                                                                                              |
| Ozone                                                                                                  | Interferes at all levels.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |

<sup>1</sup> See *Optional standards and apparatus*.

<sup>2</sup> Samples treated with sodium arsenite for interferences will be hazardous waste as regulated by the Federal RCRA for arsenic (D004). Refer to the current MSDS for safe handling and disposal instructions.

## Sampling and storage

Samples must be analyzed immediately and cannot be preserved for later analysis.

A common testing error is introduced if the analyst does not obtain a representative sample. If sampling from a tap, let the water flow for at least five minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. Perform the chlorine analysis immediately.

Avoid plastic containers since these may have a chlorine demand.

Pre-treat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized water.

If sample containers are rinsed thoroughly with deionized water after use, only occasional pretreatment is necessary. A pre-treated glass BOD bottle with a ground-glass stopper makes an ideal sample container for chlorine collection.

## Treating analysis labware

Glassware used in this test must be chlorine demand-free. Fill the 100-mL mixing cylinder and sample container with a dilute solution of chlorine bleach prepared by adding 1 mL of commercial bleach to 1 liter of water. Soak in this solution at least one hour. After soaking, rinse thoroughly with deionized water and allow to dry before use. If the mixing cylinder is thoroughly rinsed with deionized water and allowed to dry after each use, only occasional pretreatment is necessary. Do not use the same mixing cylinder for Free and Total Chlorine analysis.

Treat the Pour-Thru Cell similarly with dilute bleach and let stand for several minutes. Rinse several times with deionized water.

## Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of colored reaction products, especially if the reacted solutions are allowed to remain in the cell for long periods after measurement. Remove the buildup by rinsing the cell with 5.25 N Sulfuric Acid followed by several rinsings with deionized water.

## Accuracy check

Required for accuracy check:

- Chlorine Voluette® Ampule Standard Solution, 50 to 75-mg/L  $\text{Cl}_2$
- TenSette Pipet and tips

### Standard additions method (sample spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select Options>More>Standard Additions from the instrument menu.
3. Default values for standard concentration, sample volume and spike volumes can be accepted or edited. Enter the chlorine concentration from the ampule package. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open a Chlorine Voluette® Ampule Standard Solution, 50 to 75-mg/L  $\text{Cl}_2$ .
5. Prepare three sample spikes. Use the TenSette® Pipet to add 0.3, 0.6 and 0.9 mL of standard to three 80-mL samples, respectively. Swirl gently to mix.
6. Analyze each sample spike as described in the procedure above, starting with the 0.3 mL sample spike. Accept each standard additions value. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

## Method performance

| Program | Standard                | Precision<br>95% Confidence Limits of<br>Distribution | Sensitivity<br>Concentration change<br>per 0.010 Abs change |
|---------|-------------------------|-------------------------------------------------------|-------------------------------------------------------------|
| 82      | 1.18 mg/L $\text{Cl}_2$ | 1.17–1.19 mg/L $\text{Cl}_2$                          | 0.02 mg/L $\text{Cl}_2$                                     |

## Summary of method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color which is proportional to the chlorine concentration. Test results are measured at 530 nm.

## Consumables and replacement items

### Required reagents

| Description                          | Quantity/Test | Unit   | Catalog number |
|--------------------------------------|---------------|--------|----------------|
| Free Chlorine Reagent Set, includes: | —             | —      | 2556900        |
| DPD Indicator Powder                 | varies        | 24 g   | 2297255        |
| Free Chlorine Indicator Solution     | 1 mL          | 473 mL | 2314011        |
| Free Chlorine Buffer Solution        | 1 mL          | 473 mL | 2314111        |

### Required apparatus

| Description                                | Quantity | Unit | Catalog number |
|--------------------------------------------|----------|------|----------------|
| Cylinder, mixing graduated, 100-mL, glass  | 1        | each | 2636342        |
| Dispenser, adjustable-volume, 1.0 – 5.0 mL | 2        | each | 2563137        |
| Powder Funnel                              | 1        | each | 2264467        |

### Recommended standards

| Description                                                     | Unit   | Catalog number |
|-----------------------------------------------------------------|--------|----------------|
| Chlorine Standard Solution, Voluette® Ampule, 50-75 mg/L, 10-mL | 16/pkg | 1426810        |
| OR                                                              |        |                |
| Chlorine Standard Solution, Pour-Rite® Ampule, 50-75 mg/L, 2-mL | 20/pkg | 1426820        |
| Voluette Ampule breaker, 10 mL                                  | each   | 2196800        |
| PourRite Ampule breaker 2-mL                                    | each   | 2484600        |
| Water, deionized                                                | 4 L    | 27256          |

### Optional standards and apparatus

| Description                                         | Unit     | Catalog number |
|-----------------------------------------------------|----------|----------------|
| Pipet, TenSette® 0.1–1.0 mL                         | each     | 1970001        |
| Pipet Tips, for TenSette Pipet 1970001              | 50/pkg   | 2185696        |
| Pipet Tips, for TenSette Pipet 1970001 <sup>1</sup> | 1000/pkg | 2185628        |
| pH Paper, 0 - 14 pH range                           | 100/pkg  | 2601300        |
| Potassium Iodide, 30 g/L                            | 100 mL   | 34332          |
| Sodium Arsenite, 5 g/L                              | 100 mL   | 104732         |
| Sulfuric Acid, 1 N                                  | 100 mL   | 127032         |
| Sulfuric Acid, 5.25 N                               | 100 mL   | 244953         |

<sup>1</sup> Other sizes are available



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:  
In the U.S.A. – Call toll-free 800-227-4224  
Outside the U.S.A. – Contact the HACH office or distributor serving you.  
On the Worldwide Web – [www.hach.com](http://www.hach.com); E-mail – [techhelp@hach.com](mailto:techhelp@hach.com)

HACH COMPANY  
WORLD HEADQUARTERS  
Telephone: (970) 669-3050  
FAX: (970) 669-2932